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IS 1448-106 (1981): Methods of test for petroleum and its products, Part 106: Determination of oxidation characteristics of inhibited steam-turbine oils [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]



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Indian Standard
METHODS OF TEST FOR
PETROLEUM AND ITS PRODUCTS

[P : 106]

DETERMINATION OF OXIDATION CHARACTERISTICS
OF INHIBITED STEAM-TURBINE OILS

(First Reprint DECEMBER 1998)

UDC 621.892.098 : 543.872

*Adapted from the Joint Publication No. ASTM D 943-76/IP 157/64
(Reapproved 1975)*

1. SCOPE

1.1 This method is used to evaluate the oxidation stability of inhibited steam-turbine oils in the presence of oxygen, water, and copper and iron metals at an elevated temperature. The method may be used for testing other oils having a specific gravity less than that of water and containing rust and oxidation inhibitors.

2. OUTLINE OF THE METHOD

2.1 The oil sample is reacted with oxygen in the presence of water and an iron-copper catalyst at 95°C. The test continues until the measured total acid number of the oil is 2.0 mg KOH/g or above. The number of test hours required for the oil to reach 2.0 mg KOH/g is the 'oxidation lifetime'.

3. SIGNIFICANCE

3.1 This method is widely used and is considered valuable in estimating the oxidation stability of lubricants, especially those that are prone to

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water contamination. It should be recognized, however, that correlation between results of this method and the oxidation stability of a lubricant in field service may vary markedly with field service conditions and with various lubricants.

NOTE — Furthermore, in the course of testing a lubricant by this method, other signs of deterioration, such as sludge formation or catalyst coil corrosion, may appear which are not reflected in the calculated oxidation lifetime. The committee responsible for this method is investigating the application of alternative criteria for evaluation of lubricants using this test method.

4. APPARATUS

4.1 Oxidation Cell — of borosilicate glass, as shown in Fig. 1, consisting of a test tube, condenser, and oxygen delivery tube. The test tube has a calibration line at 300 ml (maximum error 1 ml). This calibration applies to the test tube alone at 20°C.

4.2 Heating Bath — thermostatically controlled, capable of maintaining the oil sample in the oxidation cell at a temperature of $95 \pm 0.2^\circ\text{C}$, fitted with a suitable stirring device to provide a uniform temperature throughout the bath and large enough to hold the desired number of oxidation cells immersed in the heating bath to a depth of 390 ± 10 mm and in the heating liquid itself to a depth of 355 ± 10 mm.

4.3 Flowmeter — with a capacity of at least 3 litres/h and an accuracy of ± 0.1 litre/h.

4.4 Heating Bath Thermometer* — a suitable thermometer of the following specifications:

Temperature range	72° to 126°C
Immersion, mm	100
Scale marks:	
Sub-divisions	0.2°C
Long lines at each	1°C
Numbers at each	2°C
Maximum line width, mm	0.15
Scale error, <i>Max</i>	0.2°C
Expansion chamber:	
Permit heating to	150°C
Total length, mm	390 to 400
Stem OD, mm	6.0 to 8.0
Bulb length, mm	15 to 20

*The thermometers having the designation ASTM 40C and IP 70C conforms to the above requirements.

Bulb OD, mm	Not greater than stem
Scale location:	
Bottom of bulb to line at	72°C
Distance, mm	125 to 145
Length of scale range, mm	190 to 235

4.5 Oxidation Cell Thermometer — a suitable thermometer having a range from 80 to 100°C, graduated in 0.1°C, total length 250 mm, stem diameter 6.0 to 7.0 mm, calibrated for 76 mm immersion.

4.6 Thermometer Bracket — for holding the oxidation cell thermometer, of 18-8 stainless steel, having the dimensions shown in Fig. 3. The thermometer is held in the bracket by two fluoro-elastomer O-rings of approximately 5 mm inside diameter. Alternatively thin stainless steel wire may be used.

4.7 Wire Coiling Mandrel — as shown in Fig. 2.

NOTE — The finished catalyst coil is a double spiral of copper and iron wire, 225 ± 5 mm overall length and 15.9 to 16.5 mm inside diameter. The turns of wire are evenly spaced, and two consecutive turns of the same wire as 3.96 to 4.22 mm apart, centre to centre. The mandrel shown in Fig. 2 is designed to produce such a coil. Using this mandrel, the iron wire is wound on a thread of 14.98 mm diameter, while the copper wire is wound on a thread of 15.9 mm diameter. The smaller diameter is to allow for 'spring back' of the steel wire after winding, so as to give 15.9 mm consistent inside diameter. Use of a very soft annealed steel wire may allow use of identical thread diameters for the two wires. Any arrangement that leads to the coil configuration described above is satisfactory.

4.8 Abrasive Cloth* — silicon carbide, 100 grit with cloth backing.

4.9 Syringes — glass, with Luer-Lok locking connectors, 10 and 50-ml capacities for sampling and water additions, respectively.

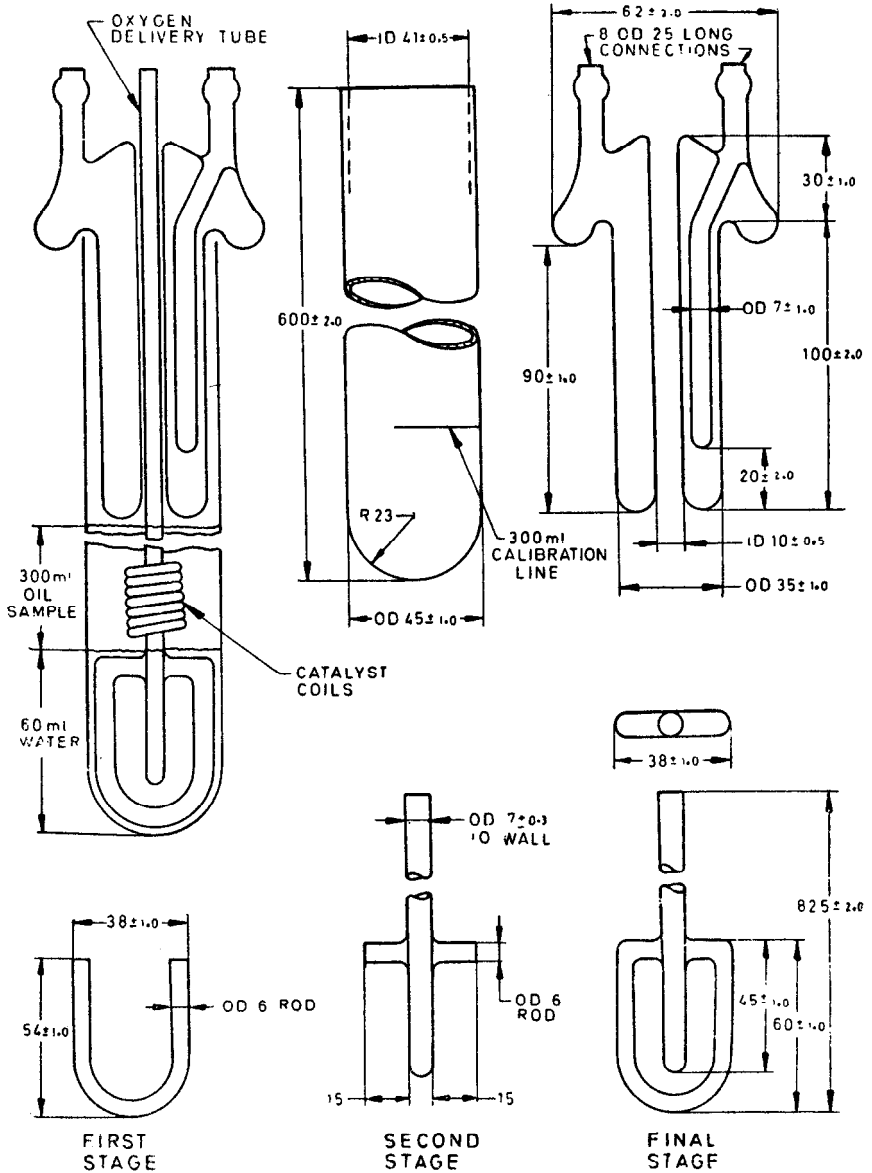
4.10 Syringe Sampling Tube† — Grade 304 stainless steel tubing 2.11 mm in outside diameter, 1.60 mm in inside diameter, 559 ± 2 mm long, with one end finished at 90° and the other end fitted with a Luer-Lok female connector. The Luer-Lok connector is preferably of elastomeric material such as polyfluorovinylchloride to provide a good seal with the syringe.

4.11 Stopper‡ — for Luer fitting of syringe sampling tube, made of polytetrafluoroethylene or polyfluorovinylchloride.

*Carborundum Co C-100-J 135F or 3 M Co Trimate 100/00 cloth, or equivalent have been found satisfactory for this purpose.

†Hamilton Co, catalogue number KF-714 syringe needles with polychlorotrifluoroethylene hub, or equivalent, have been found satisfactory for this purpose. A suitable device, such as a pipette may also be used.

‡Suitable stoppers are available from suppliers of infrared spectrometer sample cells.

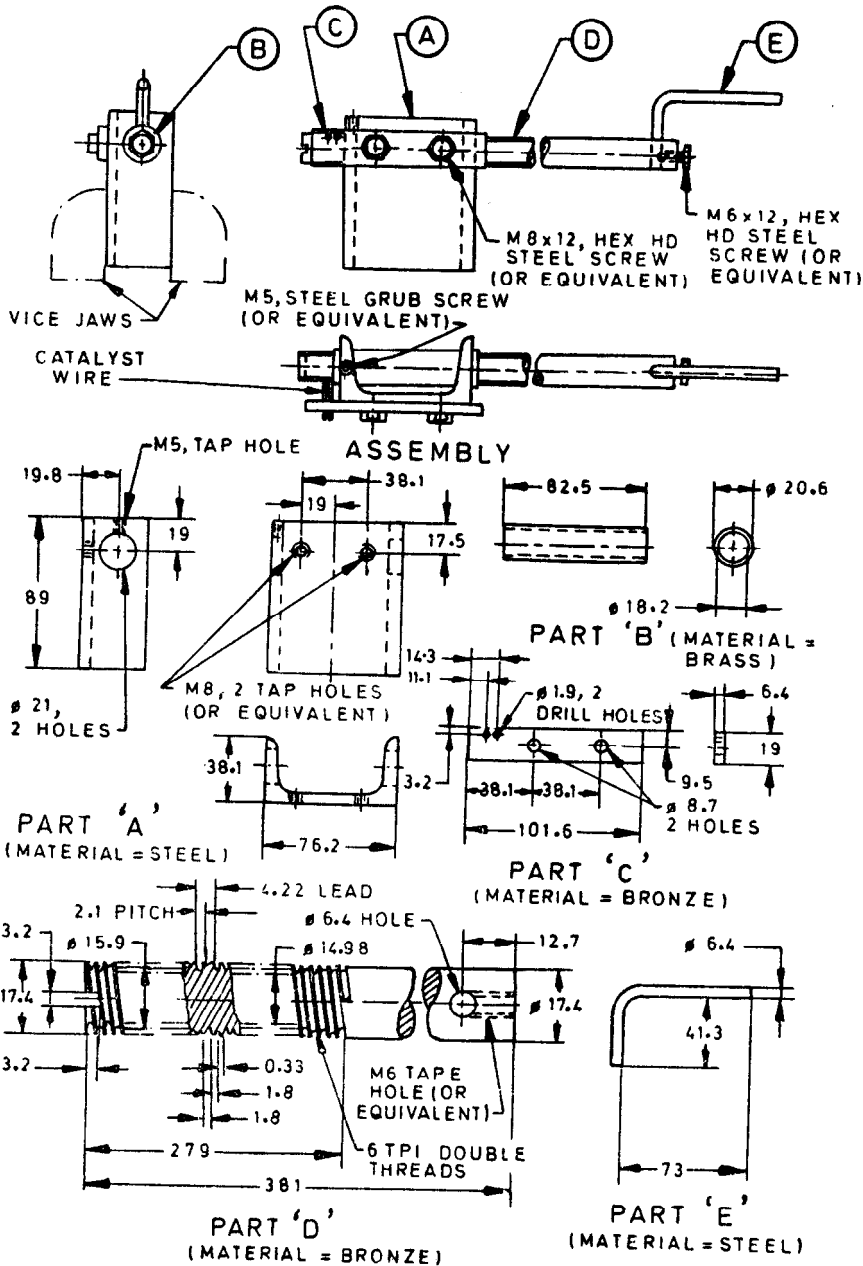


NOTE 1 — The oxidation test tube has a calibration line at 300 ml. This calibration applies to the test tube alone at 20°C.

NOTE 2 — Open tube ends to be ground and fire polished.

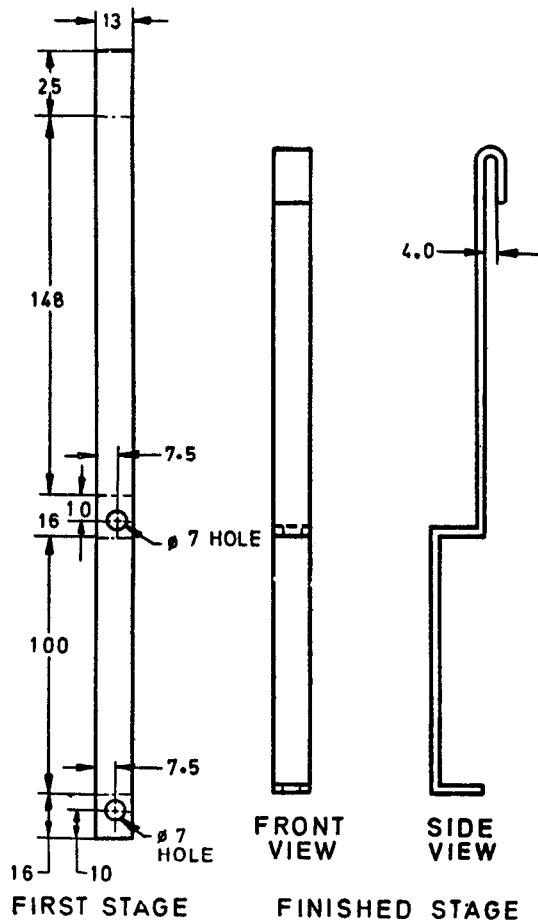
All dimensions in millimetres.

FIG. 1 OXIDATION CELL



All dimensions in millimetres.

FIG. 2 MANDREL FOR WINDING CATALYST COILS

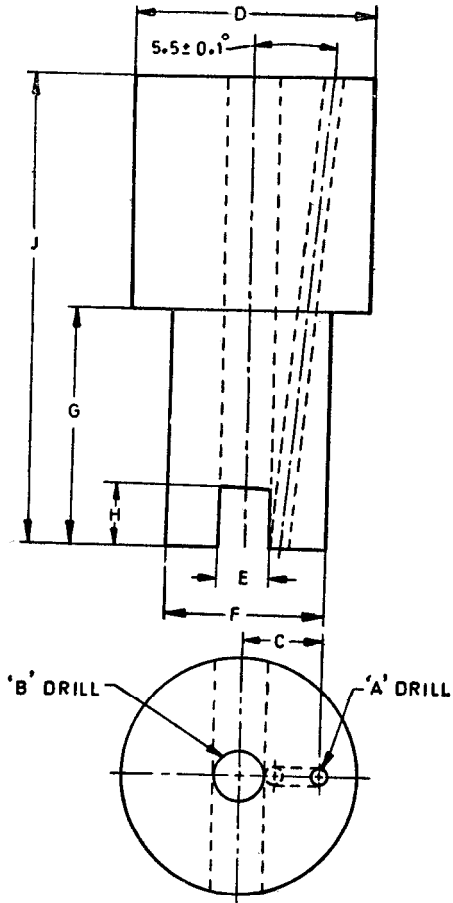


Material : 18-8 Stainless steel; 22 gauge (0.792 mm)

All dimensions in millimetres.

FIG. 3 THERMOMETER BRACKET

4.12 Sampling Tube Holder — for supporting the syringe sampling tube made of methyl methacrylate resin, having the dimensions shown in Fig. 4.



All dimensions in millimetres.

	DIMENSIONS	TOLERANCE
<i>A</i>	2.4	0.1
<i>B</i>	7.5	0.1
<i>C</i>	12.7	0.25
<i>D</i>	38.1	0.50
<i>E</i>	6.4	0.50
<i>F</i>	25.4	0.25
<i>G</i>	38.1	0.50
<i>H</i>	9.5	0.50
<i>J</i>	76.2	0.50

FIG. 4 SAMPLING TUBE HOLDER

4.13 Sampling Tube Spacer — for positioning the end of the sampling tube above the sampling tube holder, made of a length of plastic tubing polyvinyl chloride, polyethylene, polypropylene, or polytetrafluoroethylene having an inside diameter of approximately 3 mm and 51 ± 1 mm length.

4.14 Flexible Tubing — polyvinyl chloride approximately 6.4 mm in inside diameter with a wall for delivery of oxygen to the oxidation cell.

5. REAGENTS AND MATERIALS

5.1 Catalyst Wires

5.1.1 Low-Metalloid Steel Wire — 1.59 mm in diameter and conforming to the following essential requirements:

Phosphorus, percent, <i>Max</i>	0.015
Sulphur, percent, <i>Max</i>	0.05
Total of the metalloids: carbon, manganese, phosphorus, sulphur, percent, <i>Max</i>	0.14
Condition	annealed

NOTE — Other equivalent wire may be used, provided it is found satisfactory in comparative tests using this method.

5.1.2 Electrolytic Copper Wire — 1.63 mm in diameter, 99.9 percent purity. Soft copper wire of an equivalent grade may also be used.

5.2 Hydrochloric Acid, Concentrated — (relative density 1.19) technical grade (*see* IS : 265-1976*).

5.3 Acetone — 99.5 percent purity (*see* IS : 170-1976†).

5.4 n-Heptane — knock test grade, conforming to the following essential requirements:

Density at 20°C, g/ml	0.683 6 to 0.683 9
Refractive index, n _{20D}	1.387 6 to 1.387 9
Solidification temperature, °C, <i>Min</i>	— 90.72
Distillation	50 percent shall distill between 98.38 and 98.48°C. Temperature rise between 20 and 80 percent recovery shall be 0.20°C, <i>Max</i>

*Specification for hydrochloric acid (*second revision*).

†Specification for acetone (*second revision*).

5.4.1 *Naphtha, Precipitation* — conforming to the following requirements may also be used instead of *n*-Heptane:

<i>Test</i>	<i>Requirement</i>
Gravity, deg API at 15.5°C	70 to 73
Aniline point	58 to 60°C
Initial boiling point	Not less than 50°C
50 percent point	70 to 80°C
End point	Not more than 130°C

5.5 *iso*-propyl Alcohol — (see IS : 2631-1976*).

5.6 **Oxygen** — 99.0 percent minimum purity (see IS : 309-1974†), with pressure regulation adequate to maintain a constant flow of gas through the apparatus. The use of a two-stage pressure regulator on tank oxygen is recommended.

5.7 **1, 1, 1-Trichloroethane**

5.8 **Detergent** — Water soluble.

6. SAMPLING

6.1 Samples for this test shall be drawn from tanks, drums, small containers, or even operating equipment as prescribed in IS : 1447-1966‡.

7. PREPARATION OF APPARATUS

7.1 **Cleaning Catalyst** — Immediately prior to winding a catalyst coil, clean a 3.00 ± 0.01 m length of iron wire and an equal length of copper wire with wads of absorbent cotton wet with *n*-heptane and follow by abrasion with abrasive cloth until a fresh metal surface is exposed. Then wipe with dry absorbent cotton until all loose particles of metal and abrasive have been removed. In subsequent operations handle the catalyst wires with clean gloves (cotton, rubber, or plastic) to prevent contact with the skin.

7.2 **Preparation of Catalyst Coil** — Twist the iron and copper wires tightly together at one end for three turns and then wind them simultaneously alongside each other on a threaded mandrel (Fig. 2), inserting the iron wire in the deeper thread. Remove the coil from the mandrel, twist the free ends of the iron and copper wires together for three turns, and bend the twisted ends to conform to the shape of the spiral coil. The

*Specification for *isopropyl alcohol* (*first revision*).

†Specification for compressed oxygen gas (*second revision*).

‡Methods of sampling of petroleum and its products.

overall length of the finished coil should be 225 ± 5 mm. If necessary, the coil may be stretched to give the required length (*see* Note under 4.7).

7.3 Catalyst Storage — The catalyst coil may be stored in a dry, inert atmosphere prior to use. A suitable procedure for catalyst storage is given in Appendix A. Before use it should be inspected to assure that no corrosion products or contaminating materials are present. For overnight storage (less than 24 h) the coil may be stored in *n*-heptane (*see* Note).

NOTE — *n*-Heptane used for catalyst storage shall be free of traces of water and corrosive materials. Redistilled *n*-heptane conforming to 5.4 and stored in a tightly sealed bottle is suitable.

7.4 Cleaning New Glassware — Wash new oxygen delivery tubes, condensers, and test tubes with a hot detergent solution and rinse thoroughly with tap water. Clean the interiors of the test tubes, exteriors of the condensers and both interiors and exteriors of the oxygen delivery tubes with chromic acid cleaning solution (*see* Note). Rinse thoroughly with tap water until all acid is removed. Rinse all parts with distilled water and allow to dry at room temperature or in an oven. The final distilled water rinse may be followed by an *isopropyl* alcohol rinse, or acetone rinse, to hasten drying at room temperature.

NOTE — Alternatively to chromic acid cleaning solution, other suitable cleaning solutions may be used.

7.5 Cleaning Used Glassware — Immediately following termination of a test, drain the oil completely from the test tube. Rinse all the glassware with a hydrocarbon solvent to remove traces of oil, wash with a hot detergent solution using a long-handled brush, and rinse thoroughly with tap water. If deposits still adhere to the glassware, a method that has been found useful is to fill the test tubes with detergent solution, insert the oxygen delivery tubes and condensers and place the tubes in the bath at test temperature. Several hours soaking in this manner often serves to loosen all adhering deposits except iron oxide. Subsequent rinsing with hot (50°C) HCl (1+3) will serve to remove iron oxide. After all deposits are removed, rinse all glassware with chromic acid cleaning solution (*see* Note under 7.4). Rinse thoroughly with tap water until all acid is removed. Rinse all parts with distilled water and allow to dry at room temperature or in an oven. The final distilled water rinse may be followed by an *isopropyl* alcohol rinse, or acetone rinse, to hasten drying at room temperature. Store glassware in a dry dust-free condition until ready to use.

7.6 Cleaning Used Sampling Tube — Immediately following termination of a test, drain the oil completely from the sampling tube. Rinse the tube with a hydrocarbon solvent to remove traces of oil. Follow this

with a thorough rinse using 1, 1, 1-trichloroethane to remove any tenacious organic residues. Rinse with hydrocarbon solvent, for example, *n*-heptane, and blow dry with air or nitrogen.

8. PROCEDURE

8.1 Adjust the heating bath to a temperature high enough to maintain the oil in the oxidation test cell at the required temperature of $95 \pm 0.2^\circ\text{C}$.

8.2 Fill the empty oxidation test tube with 300 ml of the oil sample to the graduation mark. Slide the catalyst coil over the inlet of the oxygen delivery tube. If the wires are uneven at one end of the coil, position the coil so that this end is down. Place the oxygen delivery tube with the coil into the test tube. Place the condenser over the oxygen delivery tube and test tube. Place the sampling tube holder over the oxygen delivery tube. Insert the syringe sampling tube through the syringe sampling tube spacer and into the sampling tube holder. Position the bottom end of the sampling tube inside the catalyst coil. Insert a stopper in the Luer-Lok fitting end of the sampling tube. Immerse the test tube in the heating bath. Adjust the heating bath liquid level so that the tube is immersed in the liquid to a depth of 355 ± 10 mm. Connect the condenser to the cooling water. The temperature of the outlet water should not exceed 32°C at any time during the test.

NOTE — As an alternative to using the sampling tube holder and sampling tube spacer, the sampling tube may be secured in position by taping to the oxygen delivery tube using a suitable adhesive tape. The sampling tube is taped to the oxygen delivery tube at a distance of approximately 25 mm above the top of the condenser. The bottom of the sampling tube is positioned at 152 ± 6 mm from the bottom curved end of the oxygen delivery tube.

8.3 Connect the oxygen delivery tube to the oxygen supply (*see Caution 8.3.1*) through the flowmeter using new polyvinyl chloride flexible tubing no more than 600 mm in length. Before using, the interior of the new tubing should be rinsed with a volatile solvent (*n*-heptane) and blown dry with air. Adjust the rate of flow to 3 ± 0.1 litres and continue flow for 30 minutes; then add 60 ml of distilled water to the oxidation cell. Add the water by raising the condenser.

8.3.1 Caution — Since oxygen vigorously accelerates combustion, use the following safe handling procedures:

- i) Use oxygen at high pressure only in equipment free from oil or grease.
- ii) Keep oxygen away from combustibles, ignition sources and dirty surfaces to avoid possible ignition or explosions.
- iii) Always use a pressure regulator to deliver oxygen. Release regulator tension before opening the oxygen cylinder.

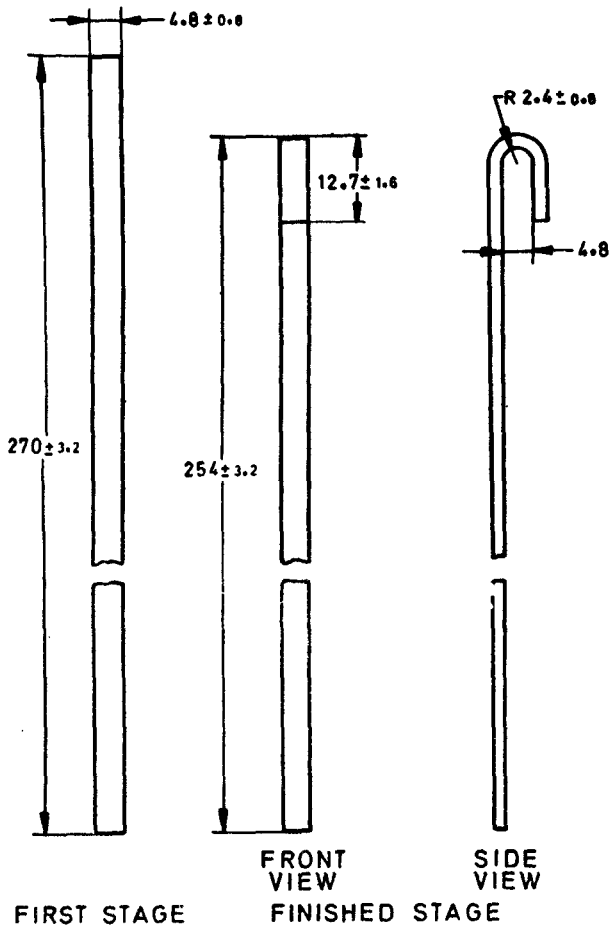
- iv) Use only equipment suitable and recommended for oxygen service.
- v) Do not allow oxygen cylinders to drop or to fall over.
- vi) Keep the oxygen cylinder valve closed when not in use.
- vii) Stand away from valve when opening oxygen cylinder.
- viii) Do not breathe or use technical grade oxygen for inhalation purpose.

8.4 Throughout the duration of the test, maintain the temperature of the oil-water mixture (sample temperature) at $95 \pm 0.2^{\circ}\text{C}$ in each test cell with oxygen flowing. Accomplish this by maintaining the bath at the temperature that is found necessary to give the required 95°C sample temperature. The temperature of the bath is always higher than the sample temperature due to the cooling effect of the oxygen gas flow and depends on heating bath medium, capacity, circulation and on the number of tests cells in the bath. Measure the sample temperature by a thermometer positioned in the oxidation cell by a thermometer bracket (see Note). Make temperature measurements only with new, undepleted, oil samples and preferably with dummy cells used specifically for temperature measurement. When an actual test sample is used, remove the thermometer immediately after temperature measurement is complete. Check the temperature in this way in various parts of a multiple-cell bath to verify uniformity of temperature control. Once the required bath temperature is found, maintain at that temperature $\pm 0.2^{\circ}\text{C}$.

NOTE — With the arrangement described in 8.4, the 76-mm immersion point of the thermometer is positioned at the oil surface. To allow for heating of the stem portion of the thermometer above the immersion point in the upper portion of the test cell, subtract 0.10°C from the thermometer reading to obtain the true test temperature.

8.5 Add additional distilled water to the oxidation cell as required, at least every 2 weeks during the test, to maintain the water level between the oxygen outlet and the shoulder of the oxygen delivery tube (see Note). Add the water using the sampling tube and the 50-ml capacity syringe.

NOTE — Under some circumstances, the level of water cannot be observed because of deposits or emulsion formation. Marking the upper oil level of the filled oxidation tube by some suitable means and maintaining this level by periodic water additions will keep the proper amount of water in the cell. The correct level for water additions may, if desired, be indicated by a movable metal strip (Fig. 5), which is clamped to the outside of the oxidation test tube by, for example, an adjustable ring-type hose clamp. To use this indicator, the lower end of the strip is set at the upper oil level when the test is started. As the test proceeds and water evaporates to cause the oil level to fall, sufficient make up water is added, particularly just before oil samples are taken, to return the oil level to the level marked by the indicator strip. After each oil sample is taken, the indicator is moved to the new oil level before continuing the test.



Material : Type 304 stainless steel; 22 gauge (0.792 mm)

All dimensions in millimetres.

FIG. 5 OIL LEVEL INDICATOR STRIP

8.6 After 500 h of test time and every 168 h (every week) thereafter, withdraw 3 ml of test oil from the oxidation test cell (see Note). This can be accomplished as follows:

8.6.1 Without interrupting the oxygen flow and without changing the position of the sampling tube, remove the sampling tube stopper and twice draw out into the syringe and return approximately 3 ml of test oil to the test cell in order to flush out the sampling tube. Then withdraw 6 ml of test oil into the syringe, hold the syringe tube vertically for 15 to 20 seconds to allow water to settle to the bottom of the syringe tube and adjust the sample size to 3 ml while holding the syringe vertically. This method allows most of the water withdrawn with the test oil to be returned to the test cell. Replace the sampling tube stopper. The 3-ml sample is dispensed into a sample vial for acid number analysis. Shake the test oil sample thoroughly before taking a sample from the vial for titration.

NOTE — Test results by this method are greatly affected by differences in sampling procedure. It is important to strictly adhere to the procedure and schedule for sampling for total acid number. An exception is where the oil is known to have a life shorter than 500 h, the sampling is at the discretion of the operator.

8.7 Discontinue the test when the measured total acid number is 2.0 mg KOH/g or above.

NOTE — The value 2.0 mg KOH/g is taken as the limiting value when testing steam-turbine oils by this method. When oxidation is proceeding rapidly in such oils, values much higher than 2.0 are frequently encountered. At that time, the state of severe oxidation of the oil, as indicated by the high total acid number, is generally confirmed by other signs of degradation such as disagreeable odour, formation of deposits, corrosion of the catalyst coil, severe darkening of the oil and emulsification of the oil-water mixture. If such signs of oxidation are noted before the scheduled time for sampling for total acid number, a sample may be taken then and the test discontinued if the measured total acid number is 2.0 mg KOH/g or above.

9. CALCULATION

9.1 Calculate the hours to 2.0 mg KOH/g total acid number (oxidation lifetime), H , as follows:

$$H = A + [(2.0 - C)/(D - C)] \times (B - A)$$

where

A = number of test hours when total acid number was last measured below 2.0,

B = number of test hours when total acid number was measured above 2.0,

C = total acid number at A hours, and

D = total acid number at B hours.

10. REPORT

10.1 Report the calculated hours to 2.0 mg KOH/g total acid number (oxidation lifetime).

11. PRECISION

11.1 The following criteria should be used for judging the acceptability of results (95 percent confidence) in the data range from 700 to 3 900 h.

11.1.1 *Repeatability* — The difference between concurrent test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials, would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

0 192 \bar{x} , where \bar{x} denotes mean value.

11.1.2 *Reproducibility* — The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would, in the long run and in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

0 332 \bar{x} , where \bar{x} denotes mean value.

NOTE — *Reproducibility with Duplicate Tests* — If two successive tests are performed by each operator in 11.1.2, the difference between the averages of the two sets of results from the tests would exceed the following values in 1 case in 20:

0 302 \bar{x} , where \bar{x} denotes the mean values of the averaged results from the two laboratories.

If more than two results are obtained by one or both of the laboratories, reproducibility can be calculated by a suitable method.

APPENDIX A

(Clause 7.3)

PROCEDURE FOR PACKAGING CATALYST COILS

A-1. MATERIALS

A-1.1 Test Tubes — borosilicate glass, 250 mm length, 25 mm outside diameter, approximately 22 mm inside diameter.

A-1.2 Caps — for test tubes, polyethylene cylindrical shape designed to closely grip outside surface of test tube.

A-1.3 Desiccant Bags — 3g silica gel granules in paper package, approximately 76 mm long, 51 mm wide, 3 mm thick.

A-1.4 Flushing Tube — stainless steel or glass, approximately 5 mm outside diameter, 305 mm long, to deliver nitrogen to bottom of test tube.

A-1.5 Nitrogen Gas — 99.7 percent minimum purity.

A-2. PROCEDURE

A-2.1 Flush a new test tube with nitrogen gas, using the flushing tube, to blow out any loose particles. The tube shall be visibly clean and dry. Hold the tube on an angle and gently slide the catalyst coil into the tube. Add a desiccant bag which has been folded lengthwise to fit in the tube. Insert the nitrogen flushing tube down the middle of the test tube, to the bottom and blow nitrogen through the tube for several seconds. Immediately after withdrawing the flushing tube, seal the test tube with a polyethylene cap.